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EDXRF DETERMINATION OF METALLIC ELEMENTS IN MINE DUST SAMPLES SUBMITTED FROM MINING RESEARCH LABORATORIES, ELLIOT LAKE

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by

C.W. Smith*

ABSTRACT

Results are presented for metallic elements ($Z > 20$) found in 130 mine airborne dust samples by energy-dispersive X-ray fluorescence spectrometry (EDXRF). These measurements were carried out at the request of a Mining Research Laboratory scientist to further characterize mine dusts that had been previously analysed as to size distribution and quartz content as part of a major study of airborne dusts in iron mines in Labrador. Aside from the major metallic components iron and manganese, only copper, zinc and lead were generally detectable at trace levels; these amounts may reflect artifacts of contamination. However, one series of samples exhibited in addition measurable levels of chromium, nickel and lead.

Calibration methodology, detection limits and precision of the measurements are discussed. Comparison to results obtained by atomic absorption spectrophotometry following total dissolution indicate that accuracies are generally better than ± 30% for amounts exceeding about 5 micrograms.

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DÉTERMINATION SFRXDE des éléments métalliques contenus des échantillons de poussière de mine fournis par les Laboratoires de recherche minière, Elliot Lake

présenté par
C.W. Smith

RÉSUMÉ

Le présent rapport donne les résultats d'une analyse spectrométrique par fluorescence aux rayons X à dispersion d'énergie, de la teneur en éléments métalliques ($z > 20$) d'échantillons de poussière d'air ambiant prélevés dans 130 mines. Les mesures ont été effectuées à la demande d'un chercheur scientifique du Laboratoire de recherche minière afin d'obtenir d'autres précisions sur les poussières de mines qui ont déjà fait l'objet d'une analyse portant sur la granulométrie des éléments et la teneur en quartz, dans le cadre d'une grande étude sur les poussières présentes dans l'air des mines de fer du Labrador. Exception faite du fer et manganèse, reconnus comme les principaux éléments métalliques, les analyses n'ont généralement permis de détecter que des traces de cuivre, de zinc et de plomb; ces traces peuvent être le fait de sources de contamination artificielles. Cependant, les échantillons d'une série contenaient, en plus, du chrome, du nickel et du plomb en quantités mesurables.

Les questions d'étalonnage, de limites des techniques de détection et de précision des mesures sont étudiées dans le rapport. La comparaison de nos résultats à ceux d'une analyse photométrique des spectres d'absorption atomique après dissolution totale des éléments montre que nos mesures conservent un degré de précision généralement supérieur à ± 30 %, dans le cas des éléments de plus de 5 microgrammes environ.

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INTRODUCTION

Results of energy dispersive X-ray fluorescence spectrometry measurements of metallic elements in 130 mine dust samples collected on silver membrane air filters are reported herein. The samples were submitted by G. Knight of the Mining Research Laboratory for research work being carried out within Minerals Research Project 320103, Mine Environment - Dust, involving evaluation of mine dusts from Labrador Iron Ores Mines (1).

EXPERIMENTAL METHODS

CALIBRATION METHODOLOGY

The calibration methodology developed has been described (2). The measurements incorporate the range of elements having sufficiently intense X-ray fluorescence lines between AgL_γ and AgK_α, i.e. elements from calcium to yttrium (K fluorescence) and lead, bismuth, thorium, and uranium (L fluorescence). For the present measurements, tungsten tube-excited molybdenum secondary target excitation was employed to increase the sensitivities over those previously obtained with a silver secondary target. Calibration standards were metallic oxides of known mass deposited on 0.8 μm-pore Ag filters, i.e. those prepared for the developmental studies. The observed sensitivities are shown in Figure 1.

DETECTION LIMIT ESTIMATES

In the absense of spectral interferences, detection limits (d.l.) are determined by counting statistics of the photopeak area (A_1). At the detection limit, this is determined by the Compton background; in the present measurements this corresponds to $A_1 = 8.6\ (Background_1)^{\frac{1}{2}}$ and $\mu g\ (d.l.) = A_1/S_1$ where S_1 is the observed sensitivity. Detection limits so derived from blank filter measurements are shown in Table 1. Since backgrounds vary from sample to sample, detection limits were computed individually for each sample. Photopeaks for Ca, Ti, V, Y, U were not observed in the samples, consequently the observed backgrounds at CrK_α and ZnK_β and established background ratios were used to estimate detection limits for these elements (to minimize data transfer).

In the presence of spectral interferences, detection limits (i.e. µg = 99% confidence uncertainty in µg) were determined from statistically propagated uncertainties.

Spectral Interferences

Between Cr and Zn, the K_β fluorescence of element Z overlaps the K_α fluorescence (i.e. the analytical signal) of element (Z+1). These interferences were removed successively using overlap parameters measured from the oxide standards. The uncertainties in the overlaps are propagated into the element uncertainties (or detection limits) reported.

Since iron is generally orders of magnitude higher in amount than most elements, its interferences predominate. The cobalt background was necessarily determined from established Co/ZnK_β background ratios. Quantitative use of the lead L_β fluorescence was precluded by iron sum-peak interferences. The titanium K_α fluorescence was interfered with by the silicon escape peak from iron.

CALCULATIONS AND UNCERTAINTY ESTIMATES

Element amounts as µg per filter and their uncertainties were computed as outlined above with a FORTRAN program (AIR83). The estimated uncertainties in the measurement parameters used are indicated in Table 2.

The statistical errors were propagated at the 95% confidence level (3).

ATOMIC ABSORPTION SPECTROPHOTOMETRY MEASUREMENTS (AAS)

Six loaded filters were dissolved and the solutions were subjected to AAS measurement of some elements as a check on the EDXRF calibration accuracy. The mylar cover was removed, rinsed in 4 mL of hot 1:1 nitric acid in a 100 mL teflon beaker and discarded. The loaded filter was transfered to the beaker, and the nitric acid was evaporated to a paste. Two mL each of water, nitric acid, 48% hydrofluoric acid and 70% perchloric acid were added and the mixture was evaporated to the appearance of perchloric acid fumes. Two mL of nitric acid and 48% hydrofluoric acid were again added, and the mixture was evaporated to a paste. The paste was dissolved in 4 mL of 2.5% nitric acid and quantitatively transfered to a 10 mL volumetric flask by successive rinsing with small volumes of this acid.

The solution was cooled and diluted to the mark. With the exception of one filter (BB49), there was no visible evidence of undissolved residue.

AAS calibration standards were similarly prepared from aliquots of standard solutions of the elements in 2.5% nitric acid and 160 mg aliquots of silver (from spectroscopy grade silver oxide), carried through the above procedure. Aliquots of the sample and standard solutions were volumetrically diluted with 2.5% nitric acid for the iron and manganese measurements.

RESULTS AND DISCUSSION

Results of the AAS measurements are compared to the corresponding EDXRF measurements in Table 3.

During the course of the AAS preparation, it was observed that the filter rims surrounding the deposit area had become contaminated with brass residue. This undoubtedly arose from physical contact with brass rings used as separators in the shipping tube. Consequently results for copper and zinc by both methods are affected by these artifacts. Following this discovery, the rim of one obviously contaminated filter was cut off and analysed by AAS separately from the loaded portion. Virtually all the copper (and zinc) are associated with the rim artifact. Comparison to the EDXRF results for the same filter indicates that the rim portion remains largely (but not completely) "unseen" by the X-ray detector, even under the condition of broad collimation employed to enhance sensitivities (Table 3). A brass separator ring was subsequently examined by EDXRF spectrometry, and found to contain only copper and zinc at observable levels.

With the exception of lead, the comparison between the EDXRF as AAS results suggests that the accuracy of the X-ray calibration is of the order ±20%, considering the potential for errors in both types of measurements (Table 3). This degree of accuracy is sufficient for screening dust samples to detect levels of elements approaching threshold limit values for dusts in workroom environments (TLV's) (4). The accuracy uncertainty for the EDXRF results exceeds the measurement precision in most cases.

The comparative results for lead are ambiguous. The agreement ranges from acceptable (D385) to poor (D387, E110), with the EDXRF results being consistently lower than the AAS values. Confirmation of the accuracy of the lead calibration consequently requires further testing involving the preparation of several lead standards on silver membrane filters.

The EDXRF results for all samples are appended. With the exception of the series D378 to D387, the results are rather unexceptional for iron mine samples; i.e. iron and manganese are observed with "trace" levels of copper and zinc (possibly brass artifacts), and sometimes traces of lead. Photopeaks due to other elements were not observed. The D300 series exhibited, in addition, detectable levels of chromium, nickel and lead.

Interpretation of the amounts found in terms of degree of dust hazard requires information on the volume of air sampled, and combination of elements and is beyond the scope of this report. Published TLV's indicate that chromium and lead represent the most serious potential hazards, as is apparent in Table 4 (4).

ACKNOWLEDGEMENTS

AAS measurements were performed by J. Hole, Ores and Fire Assay Section, Chemical Laboratory. C.A. Josling of the X-ray Fluorescence and Radiochemistry Section, Chemical Laboratory assisted with the EDXRF measurements.

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3. Lark, P.D., Craven, B.R. and Bosworth, R.C.L. "The handling of chemical data"; Oxford, Permagon Press; 1968.

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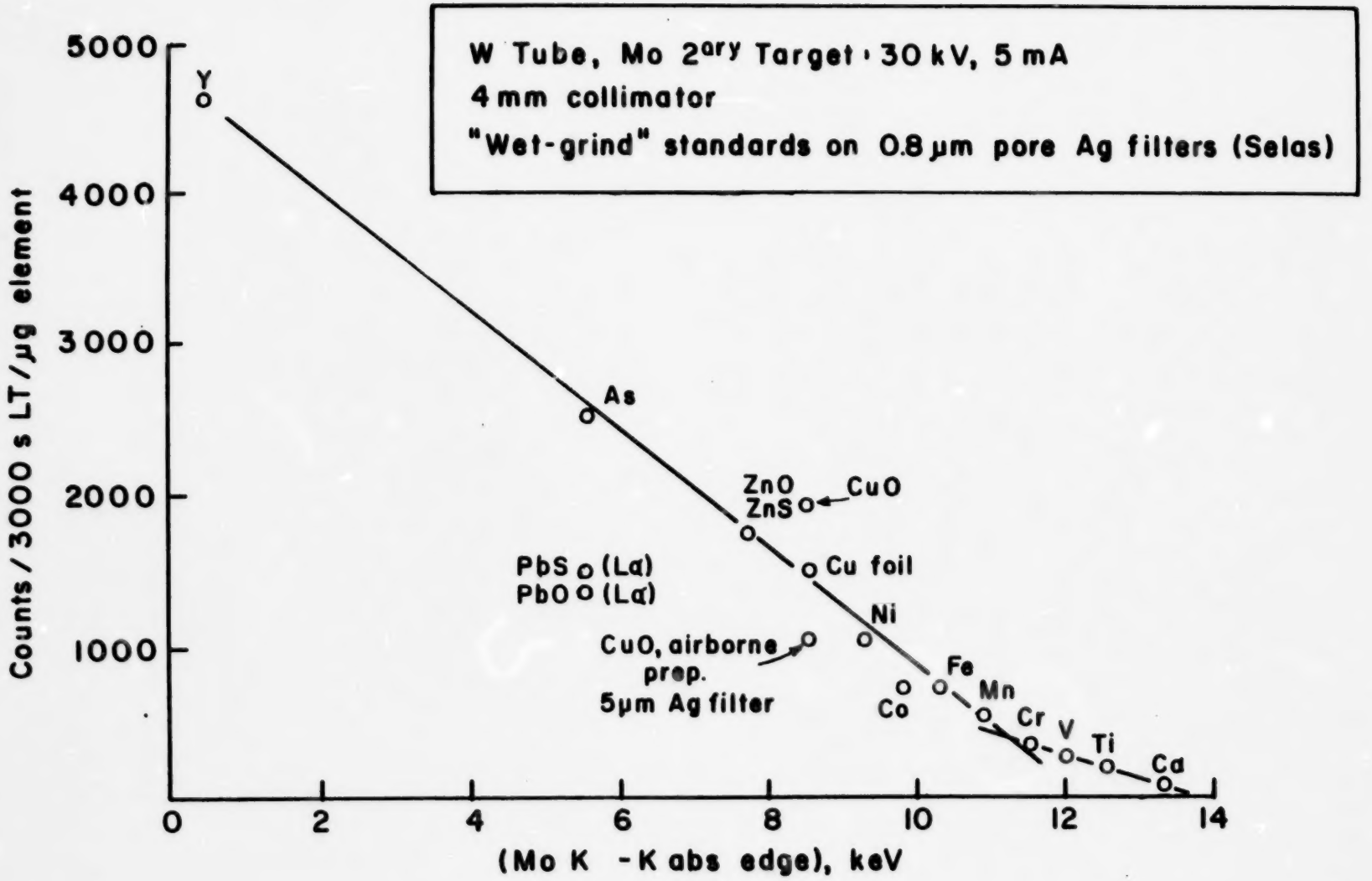
W Tube, Mo 2ary Target · 30 kV, 5 mA
4 mm collimator
"Wet-grind" standards on 0.8 µm pore Ag filters (Selas)
Counts / 3000 s LT / µg element
5000
4000
3000
2000
1000
0
2
4
6
8
10
12
14
(Mo K -K abs edge), keV
Y
As
ZnO
ZnS
CuO
PbS (Lα)
PbO (Lα)
Cu foil
Ni
CuO, airborne prep.
5µm Ag filter
Co
Fe
Mn
Cr
V
Ti
Ca

Table 1 - Detection limit estimates from observed sensitivities and counting statistics of measurements on blank 5 μm-pore size silver membrane filters. Measurement time: 3000 s (live)

Element	Detection limit, μg (99% confidence)
Ca	16
Ti	5
V	4
Cr	2.7
Mn	1.7
Fe	1.4
Co	1.2
Ni	0.9
Cu	0.6
Zn	0.6
As	0.5
Y	0.5
Pb	0.9
U	0.5

Table 2 - Calibration parameters and uncertainty estimates

1. Normalized counting reproducibility*

   Fe standard filter: 2σ = 4.6%, 12 measurements**
   Mn standard filter: 2σ = 3.8%, 10 measurements

2. Overlap; $Net_j = Integral_j - Background_j - K_{ij} * Net_i$

j	i	K_{ij}	2σ(%)	n
Mn	Cr	.151	-	1
Fe	Mn	.135	2.4	6
Co	Fe	.152	.3	12
Ni	Fe	.0004	8.	12
Ni	Co	.157		1
Cu	Ni	-	-	1
Zn	Cu	.016	35.	4

3. Background estimates; $Background_\ell = K_{\ell m} * Background_m$

ℓ	m	$K_{\ell m}$	2σ(%)	n
Ca	CrKα	2.3	13	3
Ti	CrKα	1.2	17	3
V	CrKα	1.0	10	3
Mn	CrKα	0.90	1	6
Co	ZnKβ	0.80	2	10
Ni	ZnKβ	0.81	2	10
Y	ZnKβ	4.1	5	3
U	ZnKβ	2.5	1	3

* All count data normalized to AgK_α, which minimizes the effect of geometry variation with turntable position. Results are for various turntable positions on different days, over the measurement period (3 weeks).

**Reproducibility assumed for measurement of all element reference standards; exceeds counting statistics.

Table 3 - Comparison of EDXRF and AAS results (μg found)

Sample		Cr	Mn	Fe	Ni	Cu	Zn	Pb
BB49	AAS[a]:	2.0	175	1170	1.5	14.	7.3	7.6
	XRF :	3	200	1683	3.3	4.2	2.0	4.8
E110	AAS:	2.5	6.6	803	.3	1.9	48	8.4
	XRF:	2.9	2	1057	8	1.0	5.5	.8
E252	AAS(total)[b]:	1.9	75	414	.4	2.3	71	6.2
	AAS(rim)[b] :	0.6	1.1	5	0	1.0	49	2.7
	XRF :	3	53	281	1	1.7	6.7	1.3
C180	AAS:	0.6	31	217	.4	1.5	14	2.5
	XRF:	3	27	200	1	.7	3.2	1.0
D385	AAS:	18	57	366	0.5	19.5	40	10.6
	XRF:	26	55	396	1.3	22	3.0	8.5
D387	AAS:	9.8	17	151	0	6.8	45	10.1
	XRF:	7.1	14	129	.9	7	4.6	1.1

(a) Some residue on teflon walls following digestion with HF/HNO_3 ; $HCl/HClO_4$; HF/HNO_4 .

(b) Rim, noticeably contaminated with brass, was cut off and analysed separately. Subsequent examination of other filters revealed similar contamination, consequently Cu, Zn results are above those present in the deposit. XRF collimation largely (but not completely) excludes the rim as analyte.

Table 4 - Time-weighted average threshold limit values for chemical substances in workroom air adopted by the American Conference of Governmental Industrial Hygenists for 1980 (4)

Element	Source	TLV (mg/m^3)
Ca	oxide	2
Ti	oxide	5
V	oxide, dust	0.5
Cr	chromates	0.05
Mn	compounds	5
Fe	oxide fume	5
Co	? (metal dust)	(0.1)
Ni	sulphide dust/sol. compounds	1/0.1
Cu	dusts	1
Zn	oxide, fume	5
As	sol. compounds	0.2
Pb	inorganic dusts	0.15

APPENDIX
EDXRF results for metallic elements in mine dusts

SPECTROCHEMISTRY LABORATORY CANMET

CAMPEDS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

SAMPLE	MICROGRAMS EQUIVALENT OR DETECTION LIMIT (SI)											
	CA	TI	V	CR	MN	FE	CO	NI	CU	ZN	PB	
B147	<15	< 5	< 3	< 2.5+/-***	< 1.9+/-***	402.8+/-18.6	< 1.2+/-***	< .7+/-***	1.5+/- .4	22.0+/-1.2	< .9+/-***	
B148	<15	< 5	< 3	< 2.5+/-***	< 1.8+/-***	180.6+/- 8.8	< 1.0+/-***	< .7+/-***	.8+/- .4	4.8+/- .6	< .9+/-***	
B149	<15	< 5	< 3	< 2.5+/-***	< 1.7+/-***	223.4+/-10.7	< .9+/-***	< .7+/-***	.7+/- .4	1.3+/- .5	< .9+/-***	
B150	<15	< 5	< 3	< 2.5+/-***	< 1.8+/-***	231.6+/-10.8	< 1.0+/-***	< .7+/-***	< .5+/-***	1.4+/- .5	< 1.0+/-***	
B151	<16	< 5	< 3	< 2.6+/-***	< 1.9+/-***	216.9+/-10.1	< .9+/-***	.9+/- .5	.6+/- .4	1.4+/- .5	< 1.0+/-***	
B152	<17	< 5	< 4	< 2.7+/-***	< 2.0+/-***	347.9+/-16.1	< 1.1+/-***	1.5+/- .6	.8+/- .4	3.4+/- .6	1.1+/- .8	
B153	<16	< 5	< 3	< 2.6+/-***	< 1.9+/-***	383.5+/-17.7	< 1.2+/-***	.8+/- .6	< .6+/-***	1.3+/- .5	< .9+/-***	
B154	<17	< 5	< 4	< 2.8+/-***	< 2.0+/-***	344.0+/-16.0	< 1.1+/-***	< .7+/-***	1.4+/- .5	2.5+/- .6	< 1.1+/-***	
B155	<17	< 5	< 4	< 2.7+/-***	< 2.0+/-***	286.5+/-13.6	< 1.2+/-***	1.0+/- .6	< .6+/-***	1.0+/- .6	1.2+/- .8	
B156	<18	< 5	< 4	< 2.9+/-***	< 2.2+/-***	733.5+/-33.8	< 1.6+/-***	2.1+/- .6	.8+/- .5	1.2+/- .6	< 1.2+/-***	

METHODOLOGY REFERENCE: C. SMITH AND J. DALTON, CAN. J. SPECTROSCOPY 24,163-171(1979)

REFER TO ATTACHED REPORT FOR DETAILS RE: UNCERT. AND DET. LIMIT ESTIMATES

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SPECTROCHEMISTRY LABORATORY CANMET

CAMPEDS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

SAMPLE	MICROGRAMS EQUIVQ.FOR DETECTION LIMIT (SIG)																							
	CA		TI		V		CR		MN		FE		CU		NI		CU		ZN		PB			
8827	<15	<	5	<	3	<	2.5+/-000		18.6+/-1.6		315.8+/-14.9	<	1.2+/-000		.7+/- .5		1.4+/- .4		1.0+/- .5	<	1.0+/-000			
8828	<16	<	5	<	3	<	2.6+/-000		17.7+/-1.6		285.3+/-13.6	<	1.1+/-000	<	.7+/-000		1.2+/- .4		.8+/- .5	<	.9+/-000			
8829	<15	<	4	<	3	<	2.4+/-000		7.6+/-1.2		125.3+/- 6.3		1.0+/- .7		.6+/- .4	<	.6+/-000		.9+/- .5	<	.8+/-000			
8830	<15	<	4	<	3	<	2.4+/-000		16.8+/-1.5		285.5+/-13.6		1.9+/- .9		.5+/- .4	<	.5+/-000		1.5+/- .5	<	.9+/-000			
8831	<15	<	5	<	3	<	2.5+/-000		12.7+/-1.4		160.3+/- 7.9	<	1.0+/-000		.8+/- .5	<	.5+/-000		.8+/- .5	<	.9+/-000			
8832	<16	<	5	<	4	<	2.7+/-000		31.3+/-2.1		474.8+/-22.2	<	1.4+/-000		1.2+/- .6		1.4+/- .4		1.7+/- .5	<	1.1+/-000			
8833	<17	<	5	<	4	<	2.8+/-000		35.1+/-2.3		514.6+/-24.0	<	1.5+/-000		1.2+/- .6		1.3+/- .4		1.0+/- .5		1.6+/- .8			
8834	<17	<	5	<	4	<	2.7+/-000		36.8+/-2.3		481.6+/-22.5	<	1.4+/-000		1.3+/- .6		1.2+/- .4		2.9+/- .6		1.9+/- .4			
8835	<17	<	5	<	4	<	2.9+/-000		41.2+/-2.5		472.3+/-22.1	<	1.4+/-000		2.3+/- .6		1.7+/- .5		1.5+/- .6		2.6+/- .8			
8836	<18	<	5	<	4	<	2.9+/-000		21.6+/-2.0		412.1+/-19.4	<	1.4+/-000		1.0+/- .6		1.1+/- .5		1.3+/- .6	<	1.2+/-000			

METHODOLOGY REFERENCE: C. SMITH AND J. DALTON, CAN. J. SPECTROSCOPY 24,163-171(1979)

REFER TO ATTACHED REPORT FOR DETAILS RE: UNCERT. AND DET. LIMIT ESTIMATES

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SPECTROCHEMISTRY LABORATORY CANMET

CAMPEDS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

SAMPLE	MICROGRAMS FOUND; SUB DETECTION LIMIT (SIC)											
	CA	TI	V	CR	MN	FE	CO	NI	CU	ZN	PB	
8842	<16	< 5	< 4	< 2.7+/-000	94.6+/-3.0	598.1+/-27.9	< 1.7+/-000	1.4+/- .6	1.1+/- .4	1.2+/- .5	< .9+/-000	
8843	<16	< 5	< 3	< 2.6+/-000	52.0+/-2.9	522.7+/-24.4	< 1.5+/-000	1.4+/- .6	.7+/- .4	1.3+/- .5	< 1.0+/-000	
8844	<15	< 5	< 3	< 2.5+/-000	45.2+/-2.6	444.9+/-20.8	3.3+/-1.1	1.0+/- .4	1.0+/- .4	1.2+/- .5	< .9+/-000	
8845	<17	< 5	< 4	< 2.8+/-000	66.8+/-3.6	647.5+/-30.2	< 1.7+/-000	2.0+/- .6	1.4+/- .5	1.4+/- .6	1.1+/- .8	
8846	<16	< 5	< 3	< 2.6+/-000	69.8+/-3.6	554.9+/-25.9	< 1.6+/-000	1.3+/- .6	1.5+/- .4	1.3+/- .5	< .8+/-000	
8847	<17	< 5	< 4	< 2.8+/-000	121.5+/-6.0	1088.7+/-50.3	< 2.3+/-000	2.9+/- .7	2.2+/- .4	1.7+/- .5	1.1+/- .7	
8848	<18	< 6	< 4	< 2.9+/-000	161.9+/-7.8	1421.5+/-65.6	< 2.8+/-000	3.7+/- .7	2.7+/- .4	2.0+/- .5	2.3+/- .7	
8849	<18	< 6	< 4	< 2.9+/-000	199.7+/-9.5	1683.0+/-77.7	< 3.3+/-000	4.2+/- .8	1.7+/- .4	2.0+/- .5	3.0+/- .7	
8850	<20	< 6	< 4	< 3.3+/-000	302.7+/-000	2329.4+/-0000	< 4.4+/-000	6.5+/-1.0	2.8+/- .5	2.0+/- .6	4.8+/- .9	
8851	<18	< 5	< 4	< 2.9+/-000	76.3+/-4.1	1066.6+/-49.5	< 2.4+/-000	2.6+/- .7	1.9+/- .5	1.4+/- .6	< 1.1+/-000	

METHODOLOGY REFERENCE: C. SMITH AND J. DALTON, CAN. J. SPECTROSCOPY 24,153-171(1979)

REFER TO ATTACHED REPORT FOR DETAILS RE: UNCERT. AND DET. LIMIT ESTIMATES

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SPECTROCHEMISTRY LABORATORY CANMET

CAMPEDS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

SAMPLE	MICROGRAMS EQUIVALENT (OR DETECTION LIMIT (SI))									
	CA	TI	V	CR	MN	FE	CO	NI	CU	ZN	PB
8855	<17	< 5	< 4	< 2.8+/-***	51.8+/-2.9	398.6+/-18.8	< 1.4+/-***	< .8+/-***	1.8+/- .5	1.4+/- .6	< 1.0+/-***
8857	<16	< 5	< 3	< 2.6+/-***	38.6+/-2.3	282.1+/-13.4	< 1.1+/-***	.8+/- .5	1.4+/- .4	1.1+/- .5	< 1.0+/-***
8858	<16	< 5	< 4	< 2.7+/-***	48.3+/-2.8	341.7+/-16.1	< 1.2+/-***	1.0+/- .6	1.7+/- .5	1.2+/- .5	1.1+/- .8
8859	<17	< 5	< 4	< 2.7+/-***	34.0+/-2.2	214.8+/-10.5	< 1.1+/-***	< .7+/-***	1.8+/- .5	1.7+/- .6	1.5+/- .8
8860	<17	< 5	< 4	< 2.7+/-***	74.3+/-3.9	507.4+/-23.7	< 1.5+/-***	1.2+/- .6	2.1+/- .4	1.9+/- .5	2.9+/- .7
8861	<18	< 5	< 4	< 2.9+/-***	96.9+/-4.9	638.4+/-29.8	< 1.7+/-***	1.5+/- .6	1.8+/- .5	2.0+/- .6	3.2+/- .8
8862	<17	< 5	< 4	< 2.8+/-***	97.5+/-4.9	641.0+/-29.9	< 1.7+/-***	1.5+/- .6	1.8+/- .4	2.9+/- .5	4.5+/- .7
8863	<19	< 6	< 4	< 3.1+/-***	125.0+/-6.2	776.3+/-36.0	< 1.9+/-***	1.6+/- .7	3.2+/- .5	4.1+/- .6	8.1+/- .9
8864	<16	< 5	< 3	< 2.6+/-***	56.2+/-3.1	461.6+/-21.6	< 1.4+/-***	.9+/- .6	.7+/- .4	1.5+/- .5	< .9+/-***

METHODOLOGY REFERENCE: C. SMITH AND J. DALTON, CAN. J. SPECTROSCOPY 24,163-171(1979)

REFER TO ATTACHED REPORT FOR DETAILS RE: UNCERT. AND DET. LIMIT ESTIMATES

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SPECTROCHEMISTRY LABORATORY CANMET

CAMPEOS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

SAMPLE	MICROGRAMS FOUND; SUB-DETECTION LIMIT (SIC)											
	CA	TI	V	CR	MN	FE	CO	NI	CU	ZN	PB	
8865	<17	< 5	< 4	< 2.7+/-000	30.2+/-2.0	172.7+/- 8.5	< 1.0+/-000	1.0+/- .5	.9+/- .4	1.0+/- .6	1.3+/- .8	
8866	<15	< 5	< 3	< 2.5+/-000	21.9+/-1.7	126.0+/- 6.4	< .9+/-000	< .7+/-000	.8+/- .4	.7+/- .5	< .9+/-000	
8867	<15	< 5	< 3	< 2.5+/-000	22.1+/-1.7	126.4+/- 6.4	< .9+/-000	< .7+/-000	< .6+/-000	.9+/- .5	< .9+/-000	
8869	<16	< 5	< 3	< 2.6+/-000	25.3+/-1.9	143.6+/- 7.2	< .9+/-000	< .7+/-000	.6+/- .4	1.1+/- .5	< .9+/-000	
8869	<17	< 5	< 4	< 2.8+/-000	18.4+/-1.7	99.0+/- 5.3	< .9+/-000	< .7+/-000	.8+/- .5	1.2+/- .6	< 1.0+/-000	
8870	<16	< 5	< 3	< 2.7+/-000	47.1+/-2.7	243.9+/-11.7	< 1.1+/-000	< .7+/-000	< .6+/-000	1.2+/- .5	< .9+/-000	
8871	<16	< 5	< 3	< 2.7+/-000	57.6+/-3.1	295.8+/-14.1	< 1.2+/-000	.7+/- .6	.8+/- .4	< .7+/-000	< .9+/-000	
8872	<16	< 5	< 4	< 2.7+/-000	70.1+/-3.7	366.8+/-17.3	< 1.3+/-000	1.1+/- .6	.7+/- .4	1.2+/- .5	< .9+/-000	
8873	<17	< 5	< 4	< 2.8+/-000	112.2+/-5.5	591.6+/-27.6	< 1.6+/-000	1.6+/- .6	.7+/- .4	1.1+/- .5	< .9+/-000	
8874	<15	< 5	< 3	< 2.5+/-000	31.7+/-2.0	184.4+/- 9.0	< 1.0+/-000	< .7+/-000	1.3+/- .4	1.1+/- .5	< .9+/-000	

METHODOLOGY REFERENCE: C. SMITH AND J. DALTON, CAN. J. SPECTROSCOPY 24,163-171(1979)

REFER TO ATTACHED REPORT FOR DETAILS RE: UNCERT. AND DET. LIMIT ESTIMATES

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SPECTROCHEMISTRY LABORATORY CANMET

CAMPEDS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

SAMPLE	MICROGRAMS EQUIV. FOR DETECTION LIMIT (SI)								
	CA	TI	V	CR	MN	FE	CO	NI	CU	ZN	PB
C172	<16	< 5	< 3	< 2.6+/-000	5.0+/-1.4	86.6+/- 4.8	< .9+/-000	< .7+/-000	< .6+/-000	.9+/- .5	< .9+/-000
C173	<15	< 5	< 3	< 2.5+/-000	6.7+/-1.2	67.4+/- 3.9	< .8+/-000	< .7+/-000	.6+/- .4	< .7+/-000	< .9+/-000
C174	<15	< 5	< 3	< 2.5+/-000	4.3+/-1.2	58.1+/- 3.5	< .8+/-000	< .7+/-000	.6+/- .4	.7+/- .5	< .9+/-000
C175	<15	< 4	< 3	< 2.4+/-000	5.6+/-1.2	66.9+/- 3.9	< .8+/-000	< .6+/-000	.5+/- .4	.7+/- .5	< .9+/-000
C176	<15	< 5	< 3	< 2.5+/-000	3.4+/-1.2	38.6+/- 2.9	< .8+/-000	< .7+/-000	.7+/- .4	< .7+/-000	< .9+/-000
C177	<17	< 5	< 4	< 2.8+/-000	10.7+/-1.6	116.5+/- 6.2	< 1.0+/-000	< .7+/-000	1.1+/- .5	1.2+/- .6	< 1.0+/-000
C178	<17	< 5	< 4	< 2.7+/-000	17.6+/-1.7	160.2+/- 8.0	< 1.0+/-000	< .7+/-000	.9+/- .5	3.8+/- .6	< 1.0+/-000
C179	<17	< 5	< 4	< 2.9+/-000	17.6+/-1.8	156.4+/- 7.8	< 1.0+/-000	1.0+/- .5	1.5+/- .5	2.6+/- .6	< 1.0+/-000
C180	<16	< 5	< 3	< 2.6+/-000	27.2+/-1.9	200.1+/- 9.7	< 1.1+/-000	< .7+/-000	1.8+/- .4	3.2+/- .5	1.0+/- .7
C181	<15	< 5	< 3	< 2.5+/-000	8.1+/-1.3	109.7+/- 5.7	< .9+/-000	< .7+/-000	< .5+/-000	1.5+/- .5	< 1.0+/-000

METHODOLOGY REFERENCE: C. SMITH AND J. DALTON, CAN. J. SPECTROSCOPY 24,163-171(1979)

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SPECTROCHEMISTRY LABORATORY CANMET

CANPEDS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

SAMPLE	CA	TI	V	CR	MN	FE	CO	NI	CU	ZN	PB
	MICROGRAMS EQUIVALENT FOR DETECTION LIMIT (ESTI										
D378	<15	< 5	< 3	4.4+/-1.8	11.3+/-1.4	90.8+/- 4.8	.9+/- .7	5.8+/- .5	2.0+/- .4	< .6+/-***	1.5+/- .7
D379	<16	< 5	< 3	5.6+/-2.0	11.4+/-1.5	92.9+/- 5.0	< .9+/-***	5.8+/- .6	2.7+/- .5	1.2+/- .5	< .9+/-***
D380	<15	< 5	< 3	6.7+/-1.9	9.9+/-1.5	83.4+/- 4.6	< .9+/-***	4.7+/- .6	2.8+/- .4	2.5+/- .5	1.5+/- .7
D381	<17	< 5	< 4	8.5+/-2.2	14.9+/-1.7	118.3+/- 6.1	< 1.0+/-***	6.6+/- .7	4.2+/- .5	4.0+/- .6	2.1+/- .8
D382	<17	< 5	< 4	7.8+/-2.2	17.6+/-1.7	126.0+/- 6.5	< 1.0+/-***	8.1+/- .7	3.9+/- .5	< .6+/-***	2.9+/- .8
D383	<17	< 5	< 4	17.3+/-2.3	29.2+/-2.7	227.8+/-11.0	< 1.1+/-***	13.0+/- .9	5.5+/- .5	.9+/- .6	4.6+/- .8
D384	<17	< 5	< 4	21.3+/-2.2	38.9+/-3.3	301.1+/-14.3	< 1.2+/-***	17.4+/-1.0	7.1+/- .6	< .7+/-***	5.6+/- .7
D385	<18	< 6	< 4	26.4+/-2.5	55.3+/-4.1	395.8+/-18.6	< 1.3+/-***	22.2+/-1.2	9.7+/- .7	2.3+/- .6	8.5+/- .7
D386	<19	< 6	< 4	51.4+/-3.4	96.8+/-7.0	686.9+/-31.9	< 1.7+/-***	39.9+/-2.0	17.0+/-1.0	< .6+/-***	15.8+/- .9
D386	<21	< 6	< 5	51.6+/-3.5	98.1+/-7.2	709.1+/-32.9	< 1.8+/-***	40.4+/-2.1	17.2+/-1.0	< .7+/-***	16.1+/- .9
D387	<16	< 5	< 3	7.1+/-2.0	13.8+/-1.6	128.9+/- 6.5	< .9+/-***	7.6+/- .6	3.3+/- .5	4.5+/- .6	1.5+/- .7
D387	<17	< 5	< 4	6.6+/-2.1	13.8+/-1.7	129.6+/- 6.6	< 1.0+/-***	6.7+/- .7	2.7+/- .5	4.6+/- .6	< 1.1+/-***

METHODOLOGY REFERENCE: C. SMITH AND J. DALTON, CAN. J. SPECTROSCOPY 24,163-171(1979)

REFER TO ATTACHED REPORT FOR DETAILS RE: UNCERT. AND DET. LIMIT ESTIMATES

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SPECTROCHEMISTRY LABORATORY CANMET

CAMPEDS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

SAMPLE	MICROGRAMS FOUND; FOR DETECTION LIMIT (1S)										
	CA	TI	V	CR	MN	FE	CO	NI	CU	ZN	PB
D573	<16	< 5	< 3	2.6+/-000	5.1+/-1.3	44.8+/- 3.2	< .9+/-000	< .7+/-000	.7+/- .4	.8+/- .5	< .1+/-000
D574	<16	< 5	< 3	2.6+/-000	4.0+/-1.2	24.3+/- 2.5	< .8+/-000	< .7+/-000	1.4+/- .4	3.3+/- .5	< .9+/-000
D575	<14	< 4	< 3	2.4+/-000	3.6+/-1.1	21.1+/- 2.2	< .7+/-000	< .6+/-000	< .6+/-000	1.9+/- .5	< .8+/-000
D576	<15	< 4	< 3	2.4+/-000	4.2+/-1.1	24.6+/- 2.3	1.4+/- .6	.8+/- .4	.7+/- .4	.9+/- .5	< .8+/-000
D577	<15	< 4	< 3	2.4+/-000	2.9+/-1.2	13.9+/- 2.2	< .8+/-000	< .6+/-000	1.3+/- .4	2.2+/- .5	< .9+/-000
D578	<14	< 4	< 3	2.4+/-000	6.9+/-1.2	45.1+/- 2.9	< .8+/-000	< .6+/-000	1.1+/- .4	3.0+/- .5	< .9+/-000
D579	<14	< 4	< 3	2.3+/-000	10.2+/-1.2	55.0+/- 3.3	1.1+/- .6	< .5+/-000	.7+/- .4	.8+/- .4	< .8+/-000

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SPECTROCHEMISTRY LABORATORY CANMET

CAMPEDS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

SAMPLE	MICROGRAMS FOUND±SUB DETECTION LIMIT (SIGMA)								
	CA	TI	V	CR	MN	FE	CO	NI	CU	ZN	PB
E1	<15	< 5	< 3	< 2.5+/-***	12.1+/-1.3	61.4+/- 3.6	< .8+/-***	< .7+/-***	1.1+/- .4	1.5+/- .5	< 1.0+/-***
E2	<15	< 4	< 3	< 2.4+/-***	21.5+/-1.6	93.5+/- 4.9	< .9+/-***	< .7+/-***	1.8+/- .4	3.0+/- .5	3.0+/- .7
E3	<14	< 4	< 3	< 2.4+/-***	6.8+/-1.2	49.7+/- 3.2	< .8+/-***	< .6+/-***	.9+/- .4	2.1+/- .5	< .8+/-***

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REFER TO ATTACHED REPORT FOR DETAILS RE: UNCERT. AND DET. LIMIT ESTIMATES

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SPECTROCHEMISTRY LABORATORY CANMET

CAMPEDS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

SAMPLE	MICROGRAMS FOUND (OR DETECTION LIMIT (SI))											
	CA	TI	V	CR	MN	FE	CO	NI	CU	ZN	PB	
E104	<16	< 5	< 3	< 2.6+/-***	< 2.0+/-***	1143.6+/-52.7	6.7+/-1.8	1.5+/- .4	.5+/- .4	1.2+/- .5	< .9+/-***	
E105	<16	< 5	< 3	< 2.6+/-***	< 2.0+/-***	924.6+/-42.6	2.8+/-1.5	1.8+/- .4	1.8+/- .4	2.7+/- .5	< .9+/-***	
E106	<16	< 5	< 3	< 2.6+/-***	< 2.0+/-***	855.6+/-39.4	3.9+/-1.4	1.9+/- .4	1.0+/- .4	2.3+/- .5	< .8+/-***	
E107	<16	< 5	< 3	< 2.6+/-***	< 2.0+/-***	890.9+/-41.1	3.7+/-1.5	< .5+/-***	.9+/- .4	4.4+/- .5	< .9+/-***	
E108	<15	< 4	< 3	< 2.4+/-***	< 1.7+/-***	147.5+/- 6.9	1.3+/- .6	< .5+/-***	1.0+/- .4	7.0+/- .6	< .8+/-***	
E109	<16	< 5	< 3	< 2.7+/-***	< 2.1+/-***	1117.3+/-51.4	2.5+/-1.7	2.3+/- .4	1.4+/- .4	2.0+/- .5	< .8+/-***	
E110	<15	< 5	< 3	< 2.9+/-1.8	< 2.0+/-***	1057.1+/-48.7	7.8+/-1.7	1.0+/- .4	2.0+/- .4	5.5+/- .5	< .8+/-***	
E111	<16	< 5	< 4	< 2.7+/-***	< 2.0+/-***	803.7+/-37.0	< 1.8+/-***	1.8+/- .6	1.7+/- .4	3.9+/- .5	< .9+/-***	
E112	<16	< 5	< 3	< 2.6+/-***	< 1.9+/-***	425.8+/-19.7	< 1.2+/-***	.9+/- .6	1.2+/- .4	3.5+/- .5	< .9+/-***	
E113	<17	< 5	< 4	3.3+/-2.1	< 2.3+/-***	2065.9+/-95.1	< 3.7+/-***	4.4+/- .9	2.1+/- .4	5.9+/- .6	< .9+/-***	

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SPECTROCHEMISTRY LABORATORY CANMET

CAMPEDS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

SAMPLE	MICROGRAMS FOUND, (OR DETECTION LIMIT (3S))									
	CA	TI	V	CR	MN	FE	CO	NI	CU	ZN	PB
E245	<15	< 5	< 3	< 2.5+/-***	37.3+/-2.2	211.5+/-10.2	< 1.0+/-***	1.1+/- .5	1.7+/- .4	6.7+/- .6	< .8+/-***
E246	<14	< 4	< 3	< 2.3+/-***	20.6+/-1.6	113.9+/- 5.8	< .9+/-***	< .7+/-***	1.6+/- .4	4.1+/- .5	< .8+/-***
E247	<15	< 4	< 3	< 2.4+/-***	20.4+/-1.6	114.0+/- 5.8	< .9+/-***	< .7+/-***	2.2+/- .4	4.1+/- .5	< .8+/-***
E248	<14	< 4	< 3	< 2.3+/-***	24.6+/-1.7	136.4+/- 6.7	< .9+/-***	< .7+/-***	1.0+/- .4	3.6+/- .5	< .9+/-***
E249	<15	< 5	< 3	< 2.5+/-***	9.8+/-1.3	53.3+/- 3.4	< .8+/-***	< .7+/-***	< .6+/-***	4.4+/- .5	< .9+/-***
E250	<16	< 5	< 4	< 2.7+/-***	43.3+/-2.5	242.6+/-11.7	< 1.1+/-***	1.3+/- .5	.9+/- .4	7.0+/- .6	< .9+/-***
E251	<16	< 5	< 4	< 2.7+/-***	49.9+/-2.8	274.0+/-13.0	< 1.1+/-***	.8+/- .6	1.0+/- .4	3.3+/- .6	1.2+/- .7
E252	<17	< 5	< 4	< 2.8+/-***	53.3+/-3.0	280.9+/-13.4	< 1.2+/-***	1.7+/- .6	5.3+/- .5	6.7+/- .7	1.3+/- .8
E253	<17	< 5	< 4	< 2.8+/-***	61.9+/-3.3	307.4+/-14.6	< 1.2+/-***	1.0+/- .6	2.4+/- .5	16.8+/-1.0	2.6+/- .4
E254	<16	< 5	< 3	< 2.6+/-***	58.5+/-3.2	369.0+/-17.4	< 1.3+/-***	1.1+/- .6	3.7+/- .5	4.7+/- .6	< .9+/-***

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SPECTROCHEMISTRY LABORATORY CANMET

CAMPEDS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

SAMPLE	MICROGRAMS FOUND FOR DETECTION LIMIT (S/I)											
	CA	TI	V	CR	MN	FE	CO	NI	CU	ZN	PB	
E292	<14	< 4	< 3	< 2.4+/-***	< 1.7+/-***	171.6+/- 8.4	2.5+/- .7	.8+/- .4	< .5+/-***	< .5+/-***	< .8+/-***	
E299	<15	< 4	< 3	< 2.4+/-***	2.4+/-1.1	165.3+/- 8.1	1.4+/- .7	< .5+/-***	1.4+/- .4	2.3+/- .5	1.3+/- .7	
E293	<14	< 4	< 3	< 2.3+/-***	< 1.6+/-***	112.2+/- 5.7	1.0+/- .7	< .5+/-***	.5+/- .4	.7+/- .5	1.2+/- .6	
E294	<15	< 5	< 3	< 2.5+/-***	< 1.7+/-***	99.1+/- 5.2	< .9+/-***	< .7+/-***	.7+/- .4	.7+/- .5	1.1+/- .7	
E295	<15	< 5	< 3	< 2.5+/-***	< 1.8+/-***	106.9+/- 5.5	< .9+/-***	< .7+/-***	.8+/- .4	1.9+/- .5	< 1.0+/-***	
E296	<16	< 5	< 3	< 2.6+/-***	< 1.8+/-***	31.0+/- 2.7	< .8+/-***	< .7+/-***	1.4+/- .4	1.9+/- .5	< .9+/-***	
E297	<16	< 5	< 3	< 2.6+/-***	< 1.8+/-***	168.9+/- 8.3	< 1.0+/-***	1.1+/- .5	1.1+/- .4	2.2+/- .5	< .9+/-***	
E298	<17	< 5	< 4	< 2.7+/-***	< 1.8+/-***	169.5+/- 8.0	< .9+/-***	< .7+/-***	.7+/- .5	1.5+/- .6	1.2+/- .8	
E299	<14	< 4	< 3	< 2.4+/-***	2.4+/-1.1	165.3+/- 8.1	1.5+/- .7	< .5+/-***	1.4+/- .4	2.3+/- .5	1.3+/- .7	
E292	<14	< 4	< 3	< 2.4+/-***	< 1.7+/-***	171.6+/- 8.4	1.4+/- .7	< .5+/-***	< .5+/-***	< .6+/-***	< .8+/-***	
E300	<15	< 4	< 3	< 2.4+/-***	6.3+/-1.2	203.1+/- 9.8	1.8+/- .8	< .5+/-***	2.9+/- .4	4.1+/- .5	3.5+/- .7	
E301	<15	< 5	< 3	< 2.5+/-***	< 1.8+/-***	399.0+/-18.5	1.2+/- .9	.6+/- .4	< .6+/-***	1.4+/- .5	< .9+/-***	

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REFER TO ATTACHED REPORT FOR DETAILS RE: UNCERT. AND DET. LIMIT ESTIMATES

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SPECTROCHEMISTRY LABORATORY CANMET

CAMPEOS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

| SAMPLE | MICROGRAMS FOUND; (OR DETECTION LIMIT (<)) | | |

SPECTROCHEMISTRY LABORATORY CANMET

CAMPEDS AIR FILTERS: METALLIC ELEMENT ANALYSIS BY EDXRF

SEPT. 1983

SAMPLE	NICROGRAMS FOUND FOR DETECTION LIMIT (SI)											
	CA	TI	V	CR	MN	FE	CO	NI	CU	ZN	PB	
E591	<18	< 6	< 4	< 2.9+/-000	< 2.4+/-000	2158.2+/-99.4	< 3.9+/-000	5.5+/- .9	.7+/- .4	< .7+/-000	< .9+/-000	
E592	<16	< 5	< 3	< 2.6+/-000	< 2.0+/-000	746.3+/-34.4	< 1.7+/-000	2.4+/- .6	.7+/- .4	.7+/- .5	< .9+/-000	
E593	<17	< 5	< 4	< 2.9+/-000	< 2.2+/-000	1099.7+/-50.7	< 2.2+/-000	2.8+/- .7	.8+/- .5	.8+/- .6	< .9+/-000	
E594	<17	< 5	< 4	< 2.8+/-000	< 2.0+/-000	387.5+/-17.9	< 1.2+/-000	1.9+/- .6	.9+/- .5	1.2+/- .6	< 1.1+/-000	
E595	<16	< 5	< 4	< 2.7+/-000	< 2.1+/-000	1003.2+/-46.3	< 2.1+/-000	2.6+/- .7	< .5+/-000	1.2+/- .5	< .9+/-000	
E596	<15	< 5	< 3	< 2.5+/-000	< 1.8+/-000	132.0+/- 6.3	< .8+/-000	.7+/- .5	.9+/- .4	.8+/- .5	< 1.0+/-000	
E597	<16	< 5	< 4	3.6+/-2.1	< 2.1+/-000	1102.8+/-50.8	< 2.2+/-000	3.0+/- .7	.9+/- .4	1.1+/- .5	< .9+/-000	
E598	<16	< 5	< 3	< 2.6+/-000	< 2.0+/-000	941.5+/-43.4	< 2.0+/-000	2.8+/- .6	< .5+/-000	.7+/- .5	< .8+/-000	
E599	<15	< 5	< 3	< 2.5+/-000	< 1.9+/-000	646.4+/-29.8	2.6+/-1.2	1.7+/- .4	1.1+/- .4	< .6+/-000	< .8+/-000	
E600	<16	< 5	< 3	< 2.7+/-000	< 2.0+/-000	911.9+/-42.0	< 2.0+/-000	1.3+/- .7	.6+/- .4	1.1+/- .5	< .9+/-000	
E600	<16	< 5	< 3	< 2.6+/-000	< 2.0+/-000	898.7+/-41.4	< 1.9+/-000	2.3+/- .6	.5+/- .4	.7+/- .5	< .9+/-000	

METHODOLOGY REFERENCE: C. SMITH AND J. DALTON, CAN. J. SPECTROSCOPY 24,163-171(1979)

REFER TO ATTACHED REPORT FOR DETAILS RE: UNCERT. AND DET. LIMIT ESTIMATES

POOR

QUALITY

ORIGINAL